



Lab Report XRF 435

S2 PICOFOX

Why Wait for Sample Prep!

Shortest Time-to-Result through TXRF

Introduction

The accurate chemical analysis of any sample requires a time-consuming procedure with a number of steps depending on the type of sample:

- Sampling, filtration, grinding, homogenization, chemical stabilization
- Digestion and/or extraction with hazardous chemicals, dilution or enrichment, pellet pressing, melting
- Instrument setup, calibration of the analytical instrument
- Measurement, data acquisition
- Data evaluation, reporting, data export, archiving

Each step raises costs for materials and labour, increases the risk of analytical errors and contaminations and requires operator training and instrument maintenance.

Simplify your sample preparation with TXRF

Total Reflection X-ray Fluorescence (TXRF) analysis is a versatile analytical method, which is suitable for the multi-element analysis of different kinds of sample types. For most analytical tasks sample digestion can be avoided and sample preparation can be reduced to a few simple steps. The operation of the instrument does not require any consumables or replacement parts. Due to a factory-built one time calibration the spectrum evaluation and quantification is fast and simple.

This report explains the easy procedures required for the preparation of different sample types. Furthermore, it highlights the short time-to-result achieved by the TXRF method.

Why Wait for Sample Prep!

Just a few steps are required for the sample preparation prior to an elemental analysis with TXRF. The preparation procedures for the most common sample types are described here in detail.

A variety of samples can be applied directly or after a simple dilution step:

- Liquids:
tap water, freshwater, beverages, urine and other body fluids, organic solvents.
- Suspensions and matrix rich liquids:
sewage, sea water, whole blood, blood serum, tissue homogenates, dyes.

Solid samples can be quantitatively analyzed after grinding and resuspending. Time-consuming

digestion with hazardous chemicals can be avoided.

- Solids, powders:
soils, sediments, tablets, polymers, food, lubricants, catalyzer, glass splinter, ashes.

Direct application of particles allows a standard-less analysis of the relative element composition.

- Particles:
Nanoparticles, contaminations, proteins, gunshot residues.

In addition, procedures for the preparation of samples like filters, wafer pieces, thin films, aerosols etc. are available on request.

Suspensions

Solid samples



- Fill powder in an agate mortar or micro mill



- Grind or mill carefully (e.g. Retsch MM400)



- Weigh about 20 to 50 mg, note exact amount

- Direct analysis of liquids, suspensions and particles
- Precalibrated instrument - simple quantification by internal standardization
- Save method - almost no hazardous chemicals needed
- Low cost of operation - no media or consumables required

Liquid samples



- Micro reaction tube with liquid sample



- Add internal standard



- Homogenize carefully



- Pipette 5 to 20 μ l on carrier



- Tube with raw suspension



- Dilute sample with distilled water



- Add internal standard



- Homogenize carefully



- Pipette 5 to 20 μ l on carrier



- Transfer quantitatively to a tube



- Suspend in 1 to 2 ml detergent solution



- Add standard



- Homogenize carefully



- Pipette 5 to 20 μ l on carrier

Particles



- Dab vacuum grease on carrier



- Pick-up some particles with (glass) rod



- Drop particles on grease

Dry
10 min

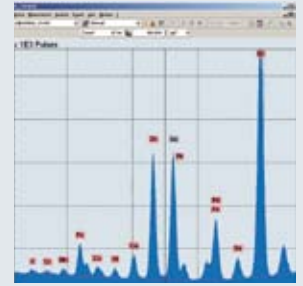


- Dry through heat or vacuum



- Load the instrument

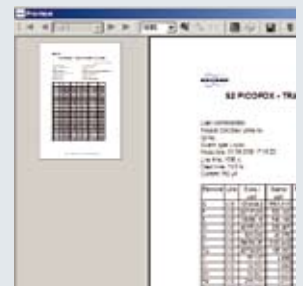
Measure
5 - 15 min



- TXRF spectrum

No	Element	Net	Conc. (µg/l)	LLD (µg/l)
1	Si	415918		
2	F	212872	90351.960	253.061
3	S	541946	112483.936	200.463
4	Cl	415041	43182.340	100.655
5	Ar	17615		
6	K	3648937	572004.830	51.545
7	Ca	861916	39669.771	29.857
8	V	3207	54.303	10.014
9	Cr	3173	30.723	7.366
10	Mn	6509	31.518	6.223
11	Fe	25299	281.943	4.303
12	Cu	7977	71.120	4.330
13	Ni	4752	44.369	3.119
14	Zn	15170	113.420	2.764
15	Zn	106723	586.666	2.401
16	Ga	107049	500.000	2.179
17	Ga	25763		
18	Au	12333	46.916	1.915
19	Au	5559		
20	Se	16227	60.017	1.901

- Table with quantitative results



- Create PDF file or print report

Element	Conc. (µg/l)	LLD (µg/l)
Si	129666.217	
F	90351.960	253.061
S	112483.936	200.463
Cl	43182.340	100.655
Ar		
K	572004.830	51.545
Ca	39669.771	29.857
V	54.303	10.014
Cr	30.723	7.366
Mn	31.518	6.223
Fe	281.943	4.303
Cu	71.120	4.330
Ni	44.369	3.119
Zn	113.420	2.764
Zn	586.666	2.401
Ga	500.000	2.179
Ga		
Au	46.916	1.915
Au		
Se	60.017	1.901

- Export data for archiving, LIMS etc.

Shorten your Time-to-Result

Effective quality and process control requires the shortest time-to-result possible. This is the time needed from sampling to the final quantitative result. Any advantage results in:

- Higher sample throughput
- Stable industrial processes due to immediate feedback
- Constant high product quality

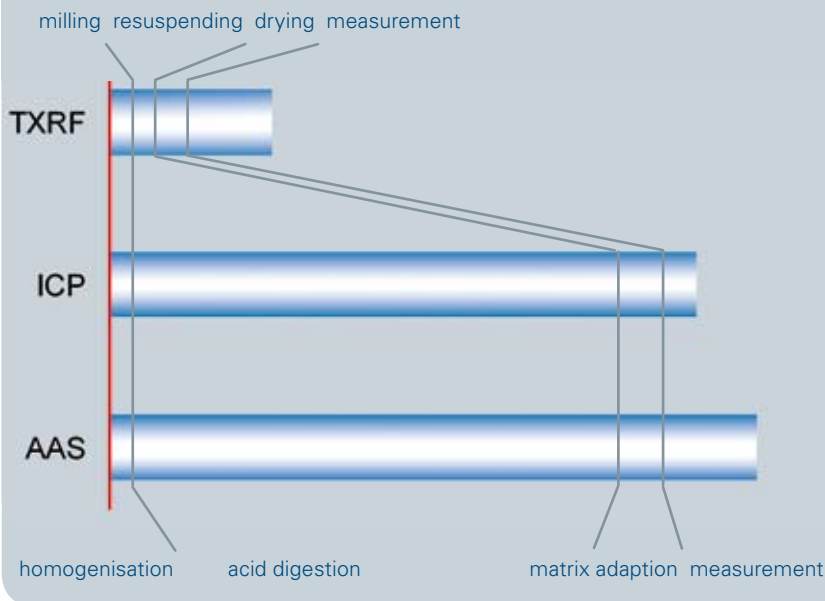
The sample preparation steps required for most common sample types prior to a TXRF analysis were shown on the previous pages.

Avoiding several preparation steps will shorten your time-to-result significantly as shown on the chart below.

- Digestion is not required for most samples types.
- The instrument is calibrated ex works, which will save up to 30% of your daily work time.
- The one-point quantification procedure with an internal standard gives accurate results automatically - effort for trainings and lab standardization will be minimized.



Comparison of the total process time for different analytical methods



In the End - Consumables

A bottomless pit

The operation of any AAS or ICP spectrometer requires the use of carrier and burning gas, standards for regular calibration and cooling water. Even at average sample throughput the costs for all consumables will sum up to about 25 % of the purchase price of an ICP-OES and 50 % of an AAS per year.

With TXRF expenses for consumables will be reduced dramatically. Inexpensive one-element standards will last for hundreds of measurements. Highest sensitivity will be achieved when using quartz sample carriers, which can be reused for more than 100 measurements. The following pictures explain the simple cleaning procedure of the carriers.

Carrier disk cleaning

- Remove sample material manually with a lint-free wipe soaked with acetone.
- Plug quartz discs in a cleaning cassette (incl. in delivery).
- Place cleaning cassette in a 1 litre glass beaker filled with 10 % nitric acid and simmer for 2 hours on a heating plate.
- Rinse the cleaning cassette with distilled water.



- Place it in a beaker filled with ultrapure water.
- Heat the beaker in a microwave oven for about 5 minutes at 800 W.
- Dry the carriers in a laboratory oven for 20 minutes at 80 °C.
- Run a blank measurement of the cleaned discs for about 180 s.
- Done! Store the clean carriers carefully without risk of contamination.



Authors

Armin Gross, Hagen Stosnach,
Bruker AXS Microanalysis GmbH, Berlin, Germany

● Bruker AXS Microanalysis GmbH

Berlin, Germany
Phone +49 (30) 670990-0
Fax +49 (30) 670990-30
info-ma@bruker-axs.de
www.bruker-axs-microanalysis.de

Bruker AXS GmbH

Karlsruhe, Germany
Phone +49 (7 21) 595-28 88
Fax +49 (7 21) 595-45 87
info@bruker-axs.de
www.bruker-axs.de

Bruker AXS Inc.

Madison, WI, USA
Phone (+1) (800) 234-XRAY
Phone (+1) (608) 276-3000
Fax (+1) (608) 276-3006
info@bruker-axs.com
www.bruker-axs.com